

WHAT IS CLAIMED IS:

1. Tantalum metal having a purity of at least about 99.995%, and an average grain size of about 125 microns or less.
2. The tantalum metal of claim 1, wherein said metal is fully recrystallized.
3. The tantalum metal of claim 1, wherein said metal is at least partially recrystallized.
4. The tantalum metal of claim 1, wherein about 98% or more of said metal is recrystallized.
5. The tantalum metal of claim 1, wherein about 80% or more of said metal is recrystallized.
6. The tantalum metal of claim 1, wherein said metal has a) a texture in which a (100) pole figure has a center peak intensity less than about 15 random or b) a log ratio of (111):(100) center peak intensities of greater than about -4.0, or c) both.
7. The tantalum metal of claim 6, wherein said center peak intensity is from about 0 random to less than about 15 random.
8. The tantalum metal of claim 6, wherein said center peak intensity is from about 0 random to about 10 random.
9. The tantalum metal of claim 6, wherein said log ratio is from greater than about -4.0 to about 15.
10. The tantalum metal of claim 6, wherein said log ratio is from about -1.5 to about 7.0.
11. The tantalum metal of claim 6, wherein said center peak intensity is from about 0 random to less than about 15 random, and said log ratio is from greater than about -4.0 to about 15.
12. The tantalum metal of claim 1 having a purity of from 99.995% to about 99.999%.
13. A metal alloy comprising the tantalum metal of claim 1.
14. A metal alloy comprising the tantalum metal of claim 6.

15. A metal alloy comprising the tantalum metal of claim 3.

16. A sputtering target comprising the tantalum metal of claim 1.

17. A sputtering target comprising the tantalum metal of claim 6.

18. A sputtering target comprising the tantalum metal of claim 3.

5 19. A capacitor can comprising the tantalum metal of claim 1.

20. A capacitor can comprising the tantalum metal of claim 6.

21. A capacitor can comprising the tantalum metal of claim 3.

22. A resistive film layer comprising the tantalum metal of claim 1.

23. A resistive film layer comprising the tantalum metal of claim 6.

10 24. A resistive film layer comprising the tantalum metal of claim 3.

25. An article comprising at least as a component the tantalum metal of claim 1.

26. An article comprising at least as a component the tantalum metal of claim 6.

27. An article comprising at least as a component the tantalum metal of claim 3.

28. Tantalum metal having a) an average grain size of about 50 microns or less, or b) a texture in
15 which a (100) pole figure has a center peak intensity equal to or less than about 15 random or c)
a log ratio of (111):(100) center peak intensities of greater than about -4.0, or combinations
SP thereof.
B2

29. The tantalum metal of claim 28 having an average grain size of from about 25 to about 50 microns.

20 30. The tantalum metal of claim 28 having a ratio of (111):(100) center peak intensities of greater than about -4.0.

31. The tantalum metal of claim 28, having both a) and b).
B

32. The tantalum metal of claim 28, wherein said metal has purity of at least 99.995% tantalum.

33. The tantalum metal of claim 28, wherein said metal has a purity of 99.999% tantalum.

34. The tantalum metal of claim 28, wherein said metal is fully recrystallized.

35. The tantalum metal of claim 32, wherein said metal is fully recrystallized.

36. The tantalum metal of claim 33, wherein said metal is fully recrystallized.

37. The tantalum metal of claim 28, wherein about 80% or more of said metal is fully recrystallized.

38. The tantalum metal of claim 28, wherein said center peak intensity is from about 0 random to about 15 random.

39. The tantalum metal of claim 28, wherein said log ratio is from about -4.0 to about 15.

40. An article comprising the tantalum metal of claim 28.

41. An article comprising the tantalum metal of claim 33.

42. A sputtering target comprising the tantalum metal of claim 28.

43. A sputtering target comprising the tantalum metal of claim 33.

44. A process for making the tantalum metal of claim 1, comprising reacting a salt containing tantalum with at least one agent capable of reducing the salt to tantalum and a second salt in a reaction container having an agitator, wherein the reaction container or a liner in the reaction container and the agitator or a liner on the agitator are made from a metal material having the same or higher vapor pressure of tantalum at the melting point of the tantalum.

45. The process of claim 44, wherein the salt containing tantalum comprises a potassium-fluoride tantalum and the agent comprises sodium.

46. The process of claim 45, wherein the second salt comprises sodium fluoride and/or sodium chloride.

5 47. The process of claim 44, wherein prior to reacting said salt containing tantalum, said process comprising forming an acid solution comprising tantalum and impurities and conducting a density separation of the acid solution containing tantalum from the acid solution containing the impurities; and crystallizing the acid solution containing the tantalum to form the salt containing tantalum.

10 48. The process of claim 47, wherein the tantalum and impurities are crushed ore comprising tantalum and impurities.

49. The process of claim 47, wherein the acid solution comprising tantalum and impurities are formed by combining acid solution with crushed ore comprising tantalum.

15 50. The process of claim 44, wherein the reaction occurs at about 800°C to about 1100°C while stirring.

51. The process of claim 44, wherein the reaction container or liner and the agitator or liner on the agitator are metal-based, wherein said metal is nickel, chromium, iron, manganese, titanium, zirconium, hafnium, vanadium, technetium, ruthenium, cobalt, rhodium, palladium, platinum, or any combination thereof.

20 52. The process of claim 51, wherein the metal is nickel or a nickel-based alloy.

53. The process of claim 51, wherein the metal is chromium or a chromium-based alloy.

54. The process of claim 51, wherein the metal is iron or an iron-based alloy.

55. The process of claim 44, further comprising recovering tantalum by dissolving the
5 second salt in an aqueous solution.

56. The process of claim 55, further comprising melting said recovered tantalum in a
sufficient vacuum to remove substantially any existing impurities in said recovered
tantalum and obtain high purity tantalum.

57. The process of claim 56, wherein the vacuum is 10^{-4} torr or more.

10 58. The process of claim 56, wherein the pressure above the melted recovered
tantalum is lower than the vapor pressures of substantially all of the impurities.

59. The process of claim 56, wherein the impurities are removed by vaporization of
the impurities.

15 60. The process of claim 56, wherein said melting is accomplished by electron beam
melting.

61. The process of claim 56, wherein said melting is accomplished by vacuum arc
remelt processing.

62. The process of claim 56, wherein the high purity tantalum is allowed to form a
solid and subjected to a rolling process, a forging process, or both.

20 63. The tantalum metal of claim 1, wherein the tantalum metal has a substantially fine
and uniform microstructure.

64. The tantalum metal of claim 1, wherein the tantalum metal has an average grain size of from about 25 to about 125 microns.

65. The tantalum metal of claim 64, wherein the tantalum metal has an average grain size of from about 25 to about 100 microns.

66. The tantalum metal of claim 65, wherein the tantalum metal has an average grain size of from about 25 to about 75 microns.

67. A process of making a sputtering target from tantalum metal having a purity of at least 99.995%, comprising:

- a) mechanically or chemically cleaning the surface of the tantalum metal, wherein the tantalum metal has a sufficient starting cross-sectional area to permit steps b) through g);
- b) flat forging the tantalum metal into at least one rolling slab, wherein the at least one rolling slab has sufficient deformation to achieve substantially uniform recrystallization after annealing in step d);
- c) mechanically or chemically cleaning the surface of the at least one rolling slab;
- d) annealing the at least one rolling slab at a sufficient temperature and for a sufficient time to achieve at least partial recrystallization of the at least one rolling slab;
- e) cold or warm rolling the at least one rolling slab in both the perpendicular and parallel directions to the axis of the starting tantalum metal to form at least one plate;
- f) flattening the at least one plate; and
- g) annealing the at least one plate to have an average grain size equal to or less than about 150 microns and a texture substantially void of (100) textural bands;

68. The process of claim 67, wherein the tantalum metal has a purity of at least 99.999%.

69. The process of claim 67, wherein the flat forging occurs after step a) and after the tantalum metal is placed in air for at least about 4 hours and from temperatures ranging from ambient to about 1200°C.

70. The process of claim 67, wherein the cold rolling is transverse rolling at ambient temperatures and the warm rolling is at temperatures of less than about 370°C.

71. The process of claim 67, wherein the annealing of the plate is vacuum annealing at a temperature and for a time sufficient to achieve recrystallization of the tantalum metal.

72. A process of making a sputtering target from tantalum metal having a purity of at least 99.995%, comprising:

- a) mechanically or chemically cleaning the surface of the tantalum metal, wherein the tantalum metal has a sufficient starting cross-sectional area to permit steps b) through i);
- b) round forging the tantalum metal into at least one rod, wherein the at least one rod has sufficient deformation to achieve substantially uniform recrystallization after annealing in step d) or step f);
- c) cutting the rod into billets and mechanically or chemically clean the surfaces of the billets;
- d) optionally annealing the billets to achieve at least partial recrystallization;
- e) axially forging billets into preforms;
- f) optionally annealing the preforms to achieve at least partial recrystallization;
- g) cold rolling the preforms into at least one plate; and
- h) optionally mechanically or chemically clean the surfaces of the at least one plate; and
- i) annealing the at least one plate to have an average grain size equal to or less than about

150 microns and a texture substantially void of (100) textural bands, wherein annealing occurs at least in step d) or f) or both.

73. The process of claim 72, wherein the tantalum metal has a purity of at least 99.999%.

5 74. The process of claim 72, wherein the round forging occurs after subjecting the tantalum metal to temperatures of about 370°C or lower.

75. The process of claim 72 wherein prior to axially the billets, the billets are annealed.

76. The process of claim 72, wherein prior to cold rolling of the preforms, the preforms are annealed.

77. The process of claim 72, wherein the annealing of the preforms is vacuum annealing at a sufficient temperature and for a time to achieve recrystallization.

78. The tantalum metal of claim 1, wherein said average grain size is from about 30 to about 125 microns.

79. The tantalum metal of claim 1, wherein said average grain size is from about 30 to about 100 microns.

80. The tantalum metal of claim 1, wherein said average grain size is about 100 microns or less.

81. The tantalum metal of claim 1, wherein said average grain size is about 50 microns or less.

82. The tantalum metal of claim 1, wherein said average grain size is from about 25 to about 100 microns.

83. Tantalum metal having a purity of at least about 99.995%, an average grain size of about 150 microns or less, and having a) a texture in which a (100) pole figure has a center peak intensity less than about 15 random or b) a log ratio of (111):(100) center peak intensities of greater than about -4.0, or c) both.

84. The tantalum metal of claim 83, wherein said metal is fully recrystallized.

85. The tantalum metal of claim 83, wherein said metal is at least partially recrystallized.
86. The tantalum metal of claim 83, wherein about 98% or more of said metal is recrystallized.
87. The tantalum metal of claim 83, wherein about 80% or more of said metal is recrystallized.
88. The tantalum metal of claim 83, wherein said center peak intensity is from about 0 random
5 to less than about 15 random.
89. The tantalum metal of claim 83, wherein said center peak intensity is from about 0 random
to about 10 random.
90. The tantalum metal of claim 83, wherein said log ratio is from greater than about -4.0 to
about 15.
- 10 91. The tantalum metal of claim 83, wherein said log ratio is from about -1.5 to about 7.0.
92. The tantalum metal of claim 83, wherein said center peak intensity is from about 0 random
to less than about 15 random, and said log ratio is from greater than about -4.0 to about 15.
93. The tantalum metal of claim 83, having a purity of from 99.995% to about 99.999%.
94. A sputtering target comprising the tantalum metal of claim 83.
95. A capacitor can comprising the tantalum metal of claim 83.
96. A resistive film layer comprising the tantalum metal of claim 83.
97. An article comprising at least as a component the tantalum metal of claim 83.
98. Tantalum metal having an average grain size of about 125 microns or less, and having 50
ppm or less metallic impurities.
99. The tantalum metal of claim 98, further having 50 ppm or less O₂, 25 ppm or less N₂, or 25
ppm or less carbon, or combinations thereof.
100. The tantalum metal of claim 98, having 10 ppm or less metallic impurities.
101. The tantalum metal of claim 100, further having 50 ppm or less O₂, 25 ppm or less N₂, or 25
ppm or less carbon, or combinations thereof.
- 25 102. The tantalum metal of claim 98, wherein said average grain size is from about 30 to about
125 microns.
103. The tantalum metal of claim 98, wherein said average grain size is about 100 microns or
less.

104. The tantalum metal of claim 98, wherein said average grain size is about 50 microns or less.

105. The tantalum metal of claim 98, wherein said average grain size is from about 25 to about 100 microns.

106. The tantalum metal of claim 98, wherein said metal is fully recrystallized.

5 107. The tantalum metal of claim 98, wherein said metal is at least partially recrystallized.

108 The tantalum metal of claim 98, wherein about 98% or more of said metal is recrystallized.

109. The tantalum metal of claim 98, wherein about 80% or more of said metal is recrystallized.

110. The tantalum metal of claim 98, wherein said metal has a) a texture in which a (100) pole figure has a center peak intensity less than about 15 random or b) a log ratio of (111):(100) center peak intensities of greater than about -4.0, or c) both.

111 The tantalum metal of claim 98, wherein said center peak intensity is from about 0 random to less than about 15 random.

112. The tantalum metal of claim 98, wherein said center peak intensity is from about 0 random to about 10 random.

113. The tantalum metal of claim 98, wherein said log ratio is from greater than about -4.0 to about 15.

114. The tantalum metal of claim 98, wherein said log ratio is from about -1.5 to about 7.0.

115. The tantalum metal of claim 98, wherein said center peak intensity is from about 0 random to less than about 15 random, and said log ratio is from greater than about -4.0 to about 15.

116. A sputtering target comprising the tantalum metal of claim 98.

117. A capacitor can comprising the tantalum metal of claim 98.

118. A resistive film layer comprising the tantalum metal of claim 98.

119. An article comprising at least as a component the tantalum metal of claim 98.

add
B10
C10
38